



Dynamic fuel retention in tokamak wall materials: An *in situ* laboratory study of deuterium release from polycrystalline tungsten at room temperature



R. Bisson^{a,*}, S. Markelj^{a,b}, O. Mourey^a, F. Ghiorghiu^a, K. Achkasov^{a,c}, J.-M. Layet^a, P. Roubin^a, G. Cartry^a, C. Grisolia^c, T. Angot^a

^a Aix Marseille Université, CNRS, PIIM UMR 7345, 13397 Marseille, France

^b Jožef Stefan Institute, Jamova cesta 39, 1000 Ljubljana, Slovenia

^c CEA, IRFM, F-13108 Saint-Paul-lez-Durance, France

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ABSTRACT

Retention of deuterium ion implanted in polycrystalline tungsten samples is studied *in situ* in an ultra-high vacuum apparatus equipped with a low-flux ion source and a high sensitivity thermo-desorption setup. Retention as a function of ion fluence was measured in the 10^{17} – 10^{21} $D^+ \cdot m^{-2}$ range. By combining this new fluence range with the literature *in situ* experimental data, we evidence the existence of a $retention \propto fluence^{0.645 \pm 0.025}$ relationship which describes deuterium retention behavior on polycrystalline tungsten on 8 orders of magnitude of fluence. Evolution of deuterium retention as a function of the sample storage time in vacuum at room temperature was followed. A loss of 50% of the retained deuterium is observed when the storage time is increased from 2 h to 135 h. The role of the surface and of natural bulk defects on the deuterium retention/release in polycrystalline tungsten is discussed in light of the behavior of the single desorption peak obtained with Temperature Programmed Desorption.

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1. Introduction

Fuel retention is one of the critical issues for plasma facing materials in fusion devices because of nuclear safety regulation related to the tritium inventory. This is the main reason why carbon plasma facing components (PFC) have been withdrawn in the final design of ITER, the international tokamak under construction in France, and tungsten has been chosen for constituting the divertor of ITER. Recently, the JET ITER-Like Wall (ILW) campaign [1] showed the benefit of tungsten and beryllium walls in terms of fuel retention as compared to the previously carbon-dominated configuration. However, the evolution of deuterium retention in the walls during operation and after discharges, i.e. the dynamic fuel retention, was different. Since in an ITER-like machine, two metals (beryllium and tungsten) with different retention behavior coexist, it is useful to perform laboratory experiments to understand

separately the retention behavior of each metal. Furthermore, such laboratory experiments are helpful to develop wall models relevant for ITER-like fusion reactor.

In the literature, few laboratory experiments focused on deuterium dynamic retention in tungsten and all of them have been performed *ex situ* [2–4], i.e. the sample has been brought to air between ion implantation and dynamic retention quantification with thermo-desorption. As it has been shown recently by Wang et al. [5], air exposure complicates these retention measurements since numerous molecular species containing deuterium are generated and retention quantification can become inaccurate. This may explain why results between *ex situ* laboratory experiments on deuterium dynamic retention are somehow inconsistent. *In situ* studies of fuel retention are thus clearly needed. Very recently, Ohno et al. [6] have introduced an *in situ* and *on-line* deuterium retention measurement method using Nuclear Reaction Analysis (NRA). They were able to determine the evolution of deuterium concentration in graphite as plasma exposure was turned on and off. It was observed, respectively, an uptake and a loss of deuterium with a time constant on the minute range. However, this powerful

* Corresponding author.

E-mail address: regis.bisson@univ-amu.fr (R. Bisson).

method may be not the best one to study the dynamic retention of deuterium because there are some concerns regarding the intrusive aspect of Nuclear Reaction Analysis. Indeed, in this method the probe beam consists of ^3He ions with kinetic energy in the MeV range, being well above most materials displacement threshold energy and defects will be created [7,8]. This could generate a probe-induced evolution of the retention properties of sample materials which would complicate the interpretation of the obtained results.

In this article, a new *in situ* deuterium implantation/thermo-desorption setup is presented which allows absolute quantification of dynamic fuel retention in tungsten samples. By using low-flux low-energy deuterium ions impinging on recrystallized tungsten samples, a fully reversible system was obtained since absolute retention for a given set of experimental parameters remained within measurement uncertainties after more than a hundred experiments on the same sample. The confidence given by their reproducibility makes these results interesting for the development of Plasma-Material Interaction models. New data sets presented here, first, extend the retention versus fluence relationship 4 orders of magnitude towards low fluences, and second, demonstrate the existence of a ~ 19 h time-scale for deuterium dynamic retention/release. While the former result could ease comparison with models on the microscopic scale, the latter result could prove important to assess fuel inventory after plasma discharges for tokamaks with ITER-like cooled divertors.

2. Material and experimental methods

2.1. Sample preparation

Polycrystalline tungsten samples were provided by A.L.M.T. Corp. with a specified 99.99 wt.% purity and cut into $10.0 \times 10.0 \times 0.4$ mm³ specimens. Samples were delivered recrystallized with a typical grain size in the ~ 30 μm range and a mirror finish obtained by mechanical polishing. They were subsequently electro-polished in our laboratory with a 2.5 wt.% NaOH solution and rinsed with distilled water. Samples were then mounted on a transfer platen made of molybdenum (purity >99.9 wt.%, ultrasonically cleaned in acetone and ethanol). Once introduced in the Ultra High Vacuum (UHV) implantation/thermo-desorption chamber (base pressure $< 4 \times 10^{-10}$ mbar), samples were subjected at least twice to a degassing procedure consisting of a linear temperature ramp of 1 K s^{-1} up to 1300 K followed by a 10 min annealing at 1300 K.

2.2. Ion implantation and sample storage

Ion implantation of deuterium in the bulk of tungsten samples were performed with a Omicron ISE 10 Sputter Ion source fed with D_2 gas (Air Liquide, N30 purity i.e. min. content of 99.9% D_2) through an all-metal leak valve. The majority deuterium ion species composing the ion beam was D_2^+ (>95%), the minority species being HD^+ , H_2^+ and D^+ , as verified with a quadrupole mass filter analyzer (Hidden EQP). Implanted ions impinged the sample with an angle of 45° and with a kinetic energy of 500 eV, which corresponds to a kinetic energy per deuteron of about 250 eV. The ion beam flux was systematically determined by measuring the current going through the tungsten sample during ion implantation with a picoammeter (Keithley 410A). The size of the implanted area was defined by a collimating aperture resulting in a 0.5 cm^2 beam spot at the sample position. The beam spot was measured using the ion-induced luminescence of a quartz sample which was recorded by a CMOS camera. The beam profile was that of a flat-top beam with an ellipsoidal elongation due to the 45° incidence angle. The typical

deuterium ion flux was $2 \times 10^{16} \text{ D}^+ \cdot \text{m}^{-2} \text{ s}^{-1}$ and therefore the sample temperature was not affected by the ion implantation. The deuterium ion fluence ($\text{D}^+ \cdot \text{m}^{-2}$) is obtained by multiplying the duration of the sample exposure to the monitored ion beam flux.

Since the ion source and the thermo-desorption oven are located in the same UHV chamber, the sample was transferred in a storage chamber (base pressure $< 1 \times 10^{-9}$ mbar) after ion implantation. Then the oven was annealed until all dissociated D_2 that eventually adsorbed on the oven was removed. Once the oven cooled down (typically >90 min), the tungsten sample was transferred back onto the oven and a thermo-desorption retention measurement could be realized.

2.3. Temperature Programmed Desorption (TPD) and retention calibration

Deuterium retention in tungsten sample is quantified using thermo-desorption. Our use of the technique is a Temperature Programmed Desorption (TPD) where the temperature of the implanted sample is linearly increased at a constant rate, while a Quadrupole Mass Spectrometer (QMS) located above the sample measures the desorption rate of molecules containing deuterium. Time integration of the QMS signal from deuterated molecular products allows quantifying the number of deuterium atoms which were present in the sample. Because TPD measurements are performed *in situ*, in the same apparatus than the ion implantation, contamination of the sample is drastically reduced and the majority of released species containing deuterium (typically $\geq 95\%$) were D_2 and HD. The remaining deuterated product was DHO and it was found to be generated from the molybdenum platen. Therefore, D_2 and HD are the only deuterated products accounted for in this work.

For TPD measurements, the sample was placed on top of the oven regulated with a PID controller (Eurotherm). Because our samples are transferable between multiple UHV chambers, we were not able to measure directly the sample temperature. Instead, we measured the temperature on the oven in the vicinity of the sample assembly with a K-type thermocouple. To obtain a linear temperature ramp on the tungsten sample, a non linear temperature ramp was programmed on the oven. The latter has been designed in the following way. In a first step, the PID controller was controlling a 1 K s^{-1} ramp on a dummy sample thanks to a second K-type thermocouple attached to it. Both the dummy sample thermocouple and the oven thermocouple temperature measurements were recorded. In a second step, the PID controller was programmed with a non-linear function (a sequence of several linear ramps) to reproduce the oven thermocouple temperature excursion observed during the 1 K s^{-1} dummy sample ramp. Running the regulation on the oven thermocouple with the designed non-linear program gave a 1 K s^{-1} temperature ramp on the dummy sample thermocouple with an excellent reproducibility. This non-linear program was used in this work to perform 1 K s^{-1} TPD ramp on tungsten samples.

In order to achieve high-sensitivity detection of desorbing species, a differentially pumped QMS (Hidden 3F/PIC) is used to collect directly the molecular species desorbing from the sample. The differential pumping chamber is home-designed and has a low background pressure ($< 8 \times 10^{-11}$ mbar). Conversion of the QMS signal into desorption rate from the sample necessitates to know the angular distribution of the released deuterium species, the geometric acceptance of the differential stage, and the QMS sensitivity for the considered species. The angular distribution of desorbing species is assumed to follow a cosine distribution. This hypothesis is based on the corrugated polycrystalline nature of the sample whose distributions of grain orientations (and cut

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